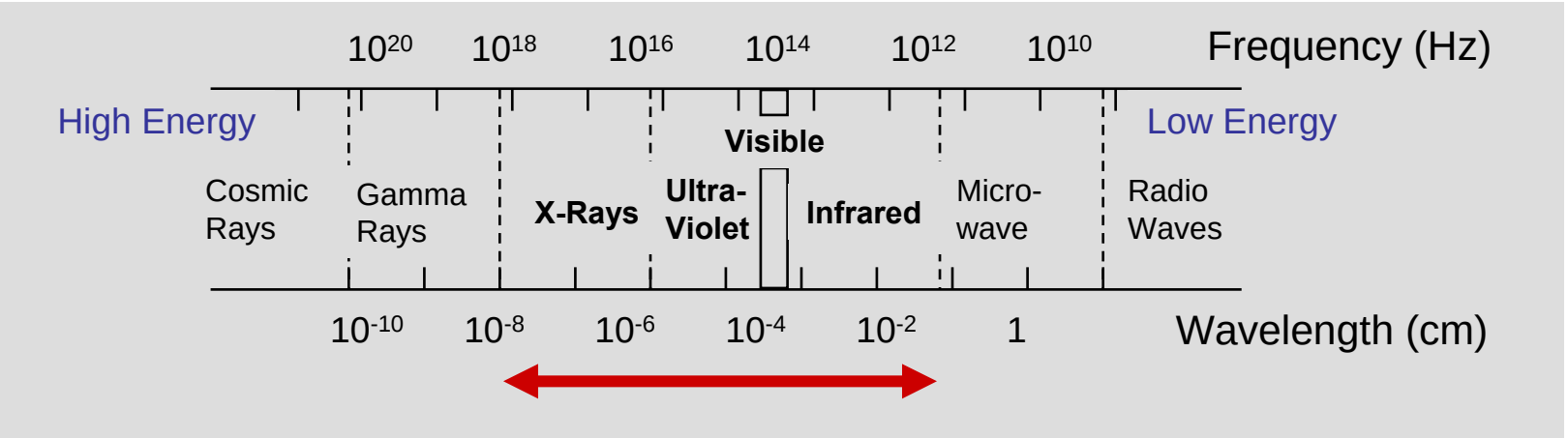
Imaging and Microspectroscopy at the NSLS

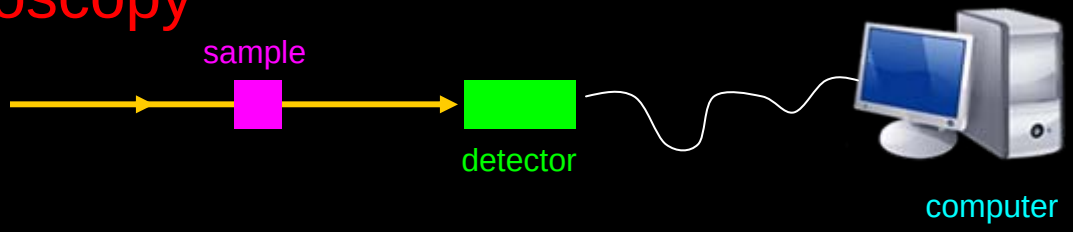


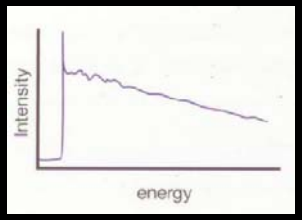
VUV-IR & X-Ray rings provide broader spectral range than any other facility in world for a wide range of techniques:

- Infrared: far- and mid-infrared microspectroscopy and imaging; potential for near-IR imaging, UV imaging
- Soft x-ray: Scanning transmission x-ray microscopy; potential for full-field x-ray microscopy
- Hard x-ray: fluorescence imaging, micro-XANES, micro-EXAFS, microtomography, and microdiffraction

Sample Interactions with Synchrotron Light

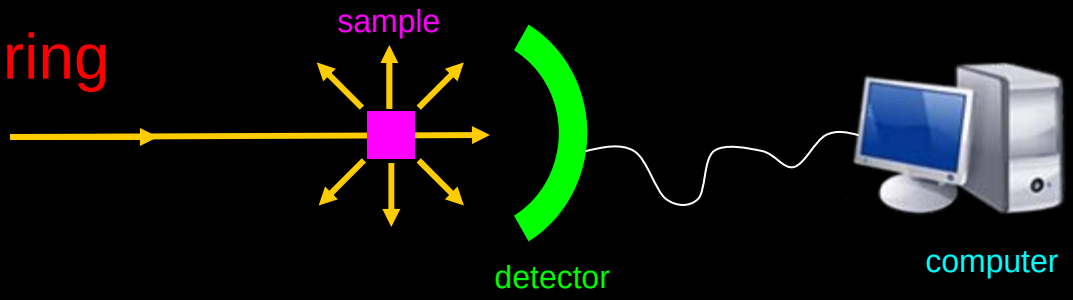
Spectroscopy

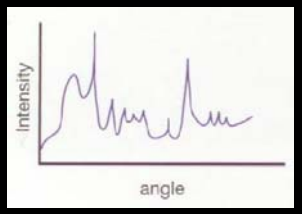




data

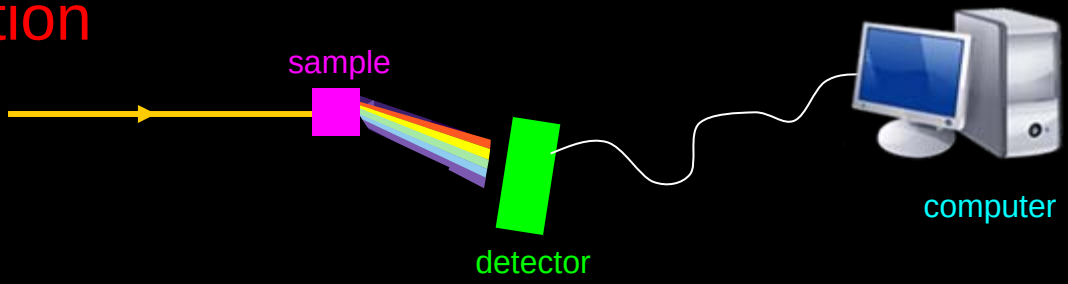
Scattering

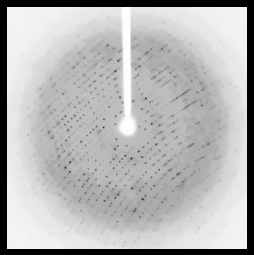




data

Diffraction

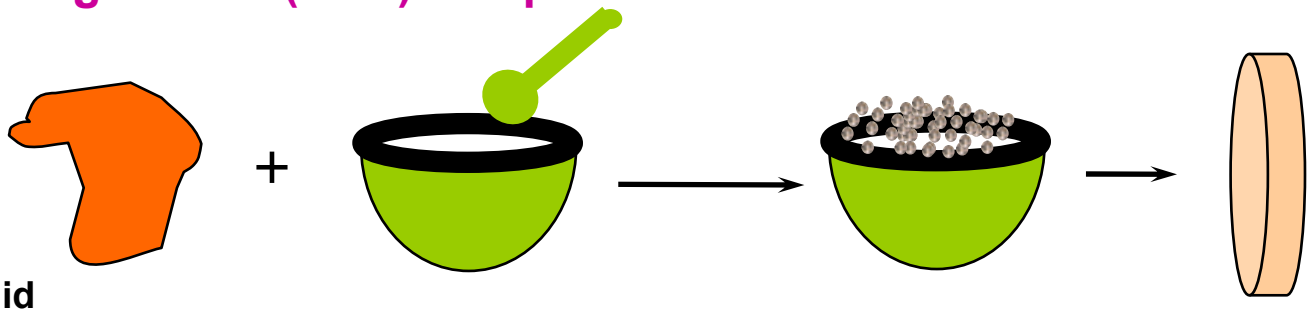




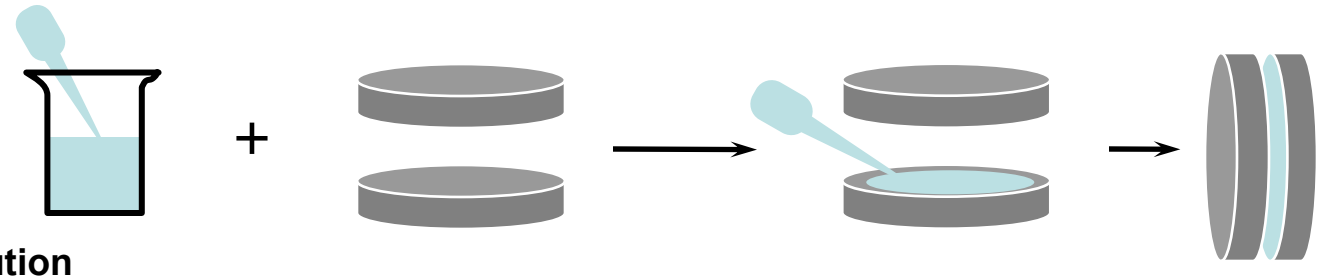
data

Bulk Methods vs. Micro-probe

Homogeneous (bulk) samples



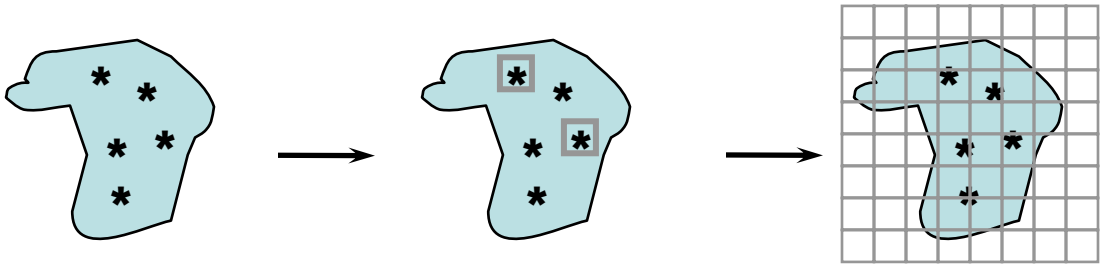
solid



solution

Spatial Resolution:
~1-10 mm

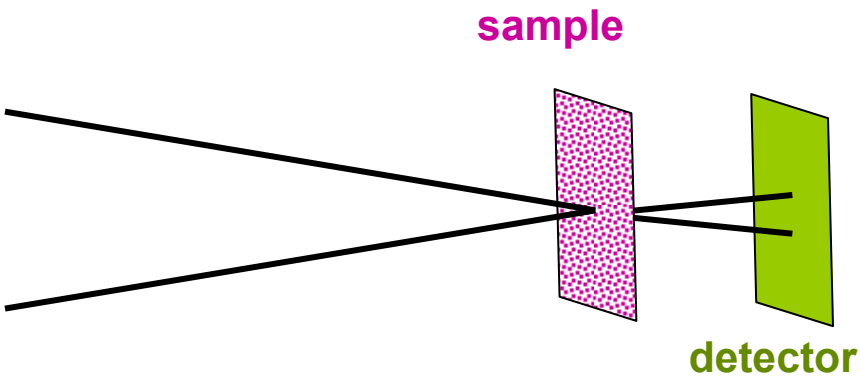
Microscopic Heterogeneity



Spatial Resolution:
~ 25 - 30 μm (globar)
~ 3 - 10 μm (synchrotron)

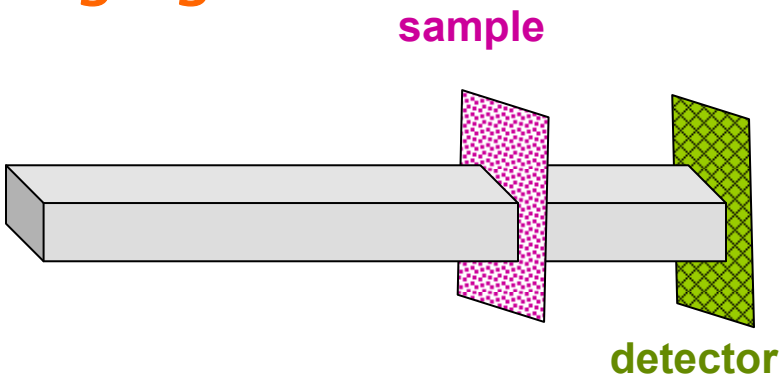
Microprobe vs. Imaging

Microprobe



- Focus beam to small spot on sample
- Raster scan sample through beam

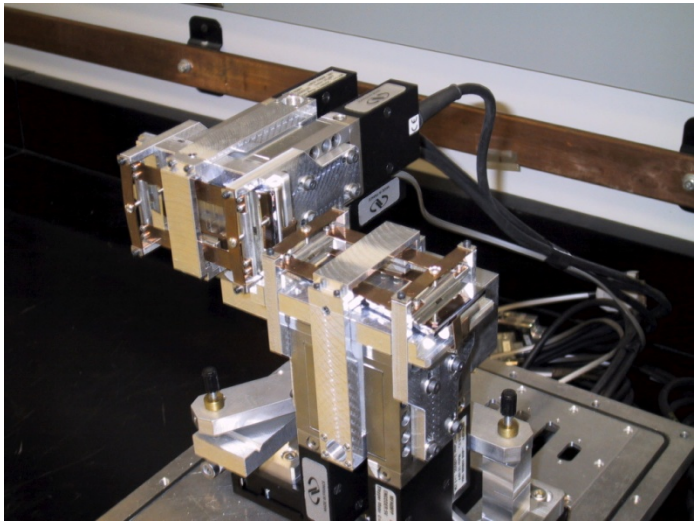
Imaging



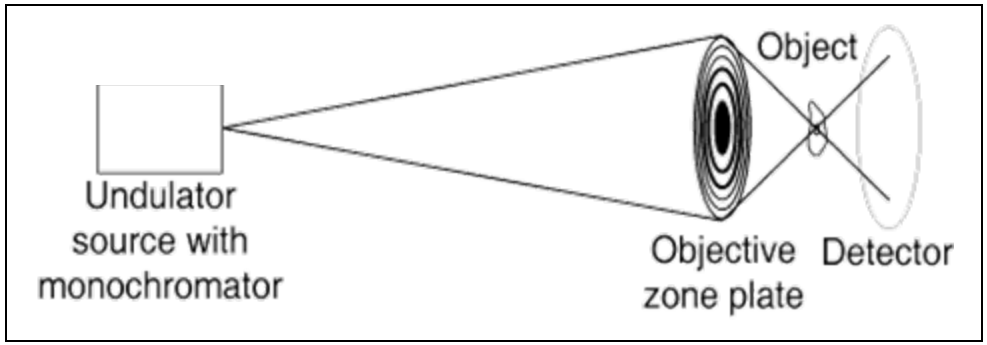
- Illuminate whole sample with big beam
- Use pixel array detector for spatial resolution

Beam Focusing

- KB Mirrors
  - focus to 5-10 microns
  - Retains most flux

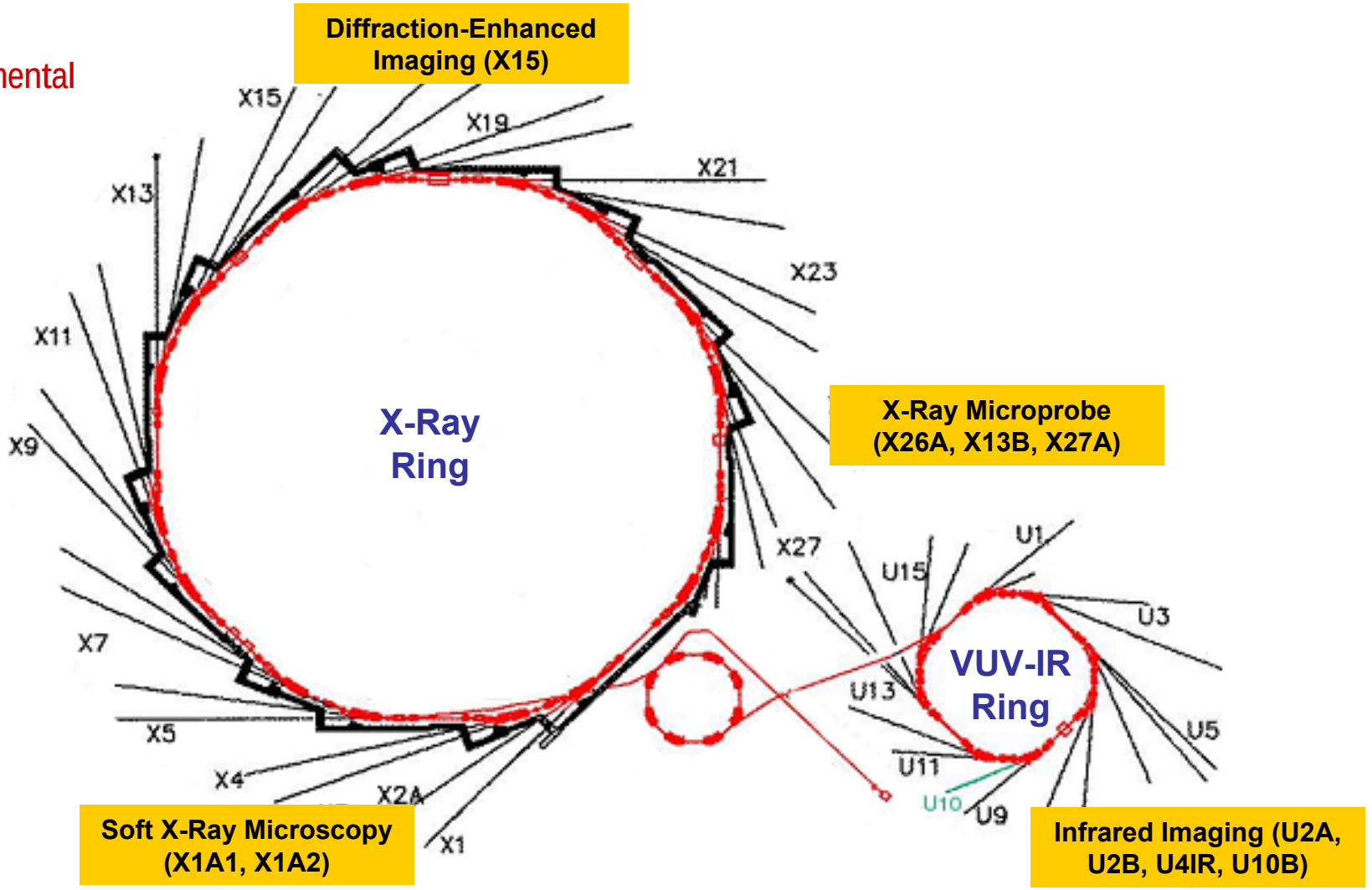


- Zone Plates
  - Focus to 30-100 nm
  - Low efficiency; need bright beam



Imaging Beamlines at the NSLS

NSLS Experimental Floor



Soft X-Ray Spectromicroscopy

ENERGY RANGE:

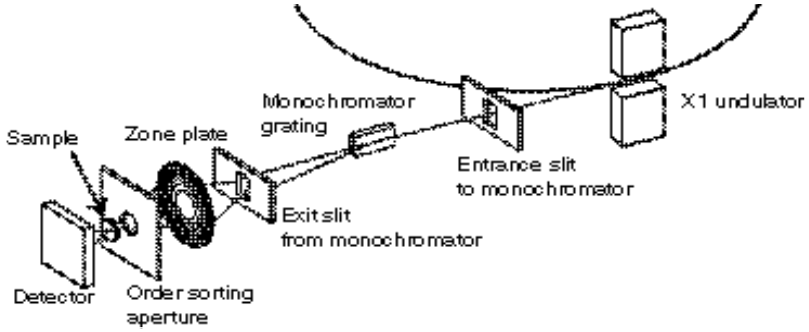
- 100 – 1000 eV

CONTRAST:

- absorption of low-Z elements, e.g. C, N, O

SPATIAL RESOLUTION:

- 30 – 50 nm



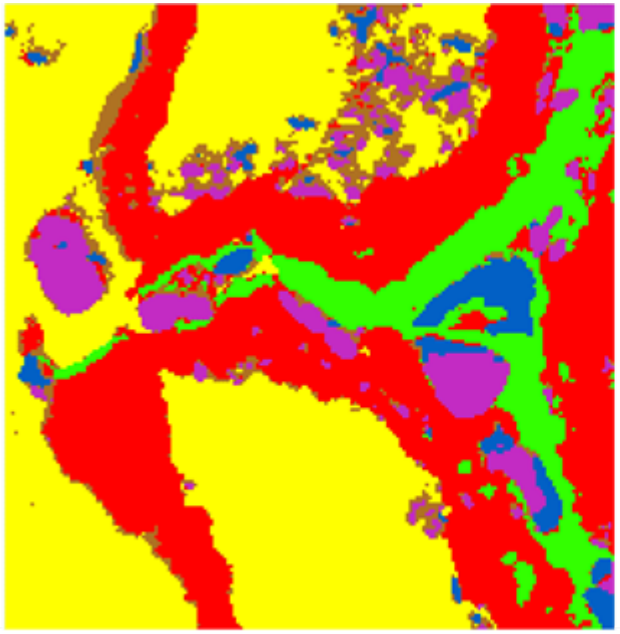
Beamline X1A: Scanning Transmission X-Ray Microscopy (STXM)

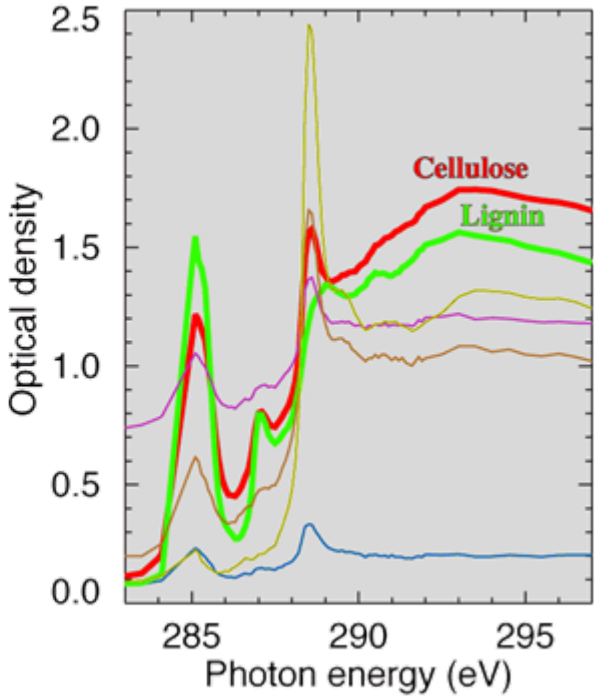
UNIQUE CAPABILITIES:

- Higher resolution than optical microscopy
- Intrinsic contrast between organic material and water
- Good penetration in micron-thick specimens.
- Sample chemistry can be probed with spectromicroscopy
- Wet and dry materials can be imaged (cryo-microscopy for hydrated samples)
- Examples: role of protamines in the packing of DNA in sperm; trabecular architecture in bone; organic carbon content in interplanetary dust particles; geochemical evolution of biomacromolecular ensembles (*e.g.* cell membranes with pressure, temperature, and time); biodegradation of chemically resistant biopolymers

Spectromicroscopy and Biofuels

- Ethanol from lignocellulose materials is promising: large fraction of total biomass, easier cultivation.
- But there are great challenges in economically separating cellulose from lignin!
- Soft x-ray spectromicroscopy can map cellulose and lignin so that one can see the effects of various enzymes.





Lignin and cellulose in 400 million year old chert: Boyce *et al.*, *Proc. Nat. Acad. Sci.* **101**, 17555 (2004), with subsequent pattern recognition analysis by Lerotic *et al.*, *Ultramicroscopy* **100**, 35 (2004).

X-Ray Fluorescence Microprobe

ENERGY RANGE:

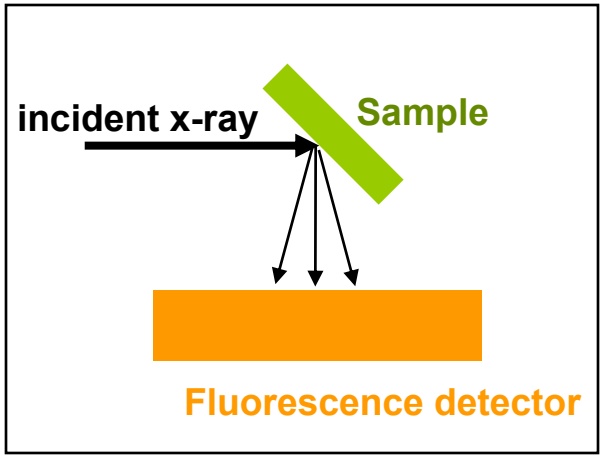
- 2 – 20 keV

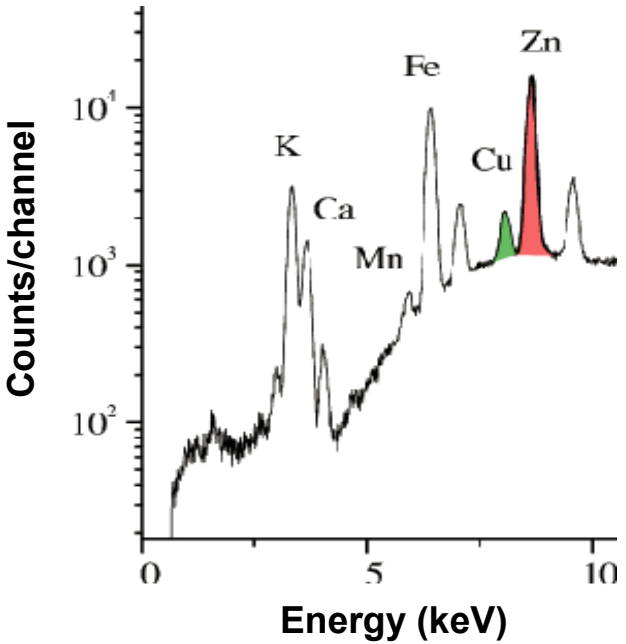
CONTRAST:

- absorption and fluorescence emission of metals

SPATIAL RESOLUTION:

- 1 – 10 μm

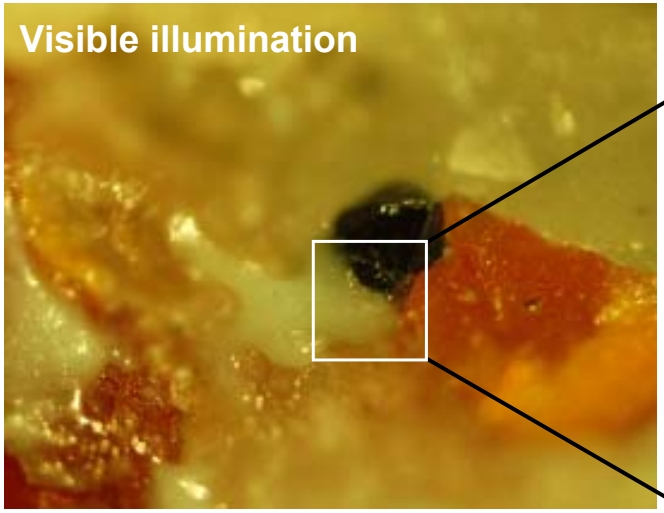


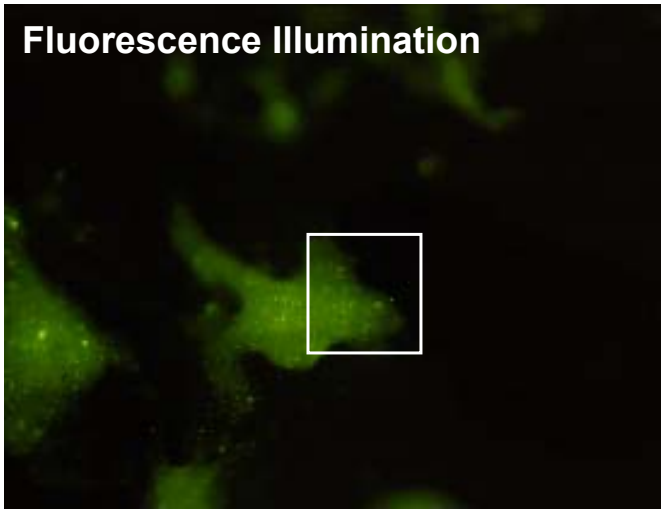


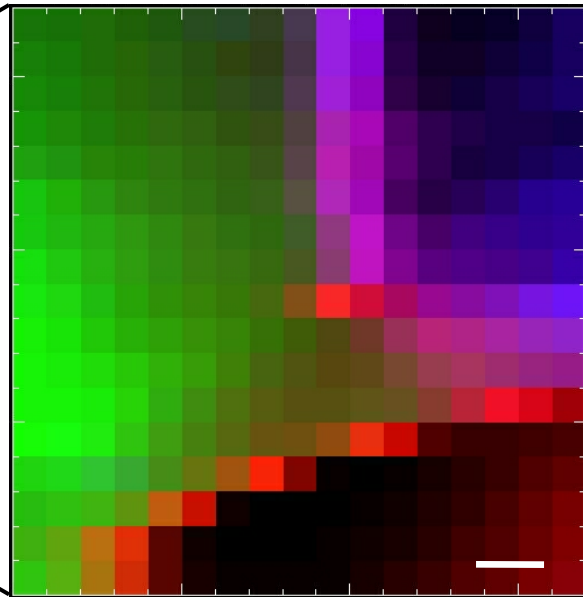
UNIQUE CAPABILITIES:

- Trace metal determination at sub-mg kg^{-1} levels in situ
- Analyses can be performed non-destructively in air on wide range of sample thicknesses and natural environment
- 3-D tomography also possible
- Examples: the role of metal ion accumulation in neurological diseases such as amylotrophic lateral sclerosis (ALS), Alzheimer's disease, scrapie; characterization of heavy metal contamination in soils; the uptake of metals in plants (e.g. phytoremediation)

Mineral Crystallite Identification







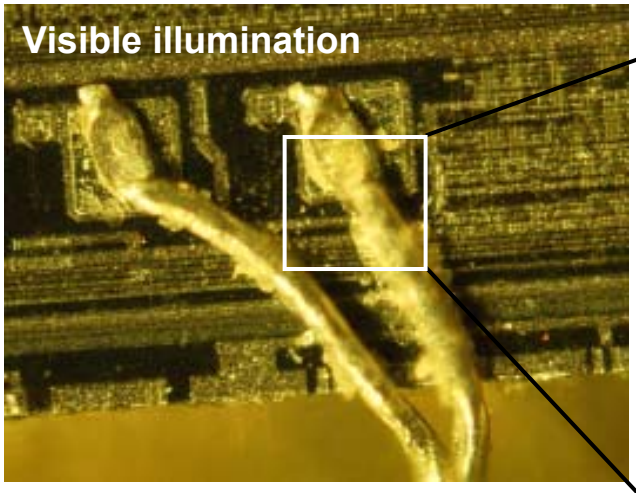
Ca calcite ($CaCO_3$)

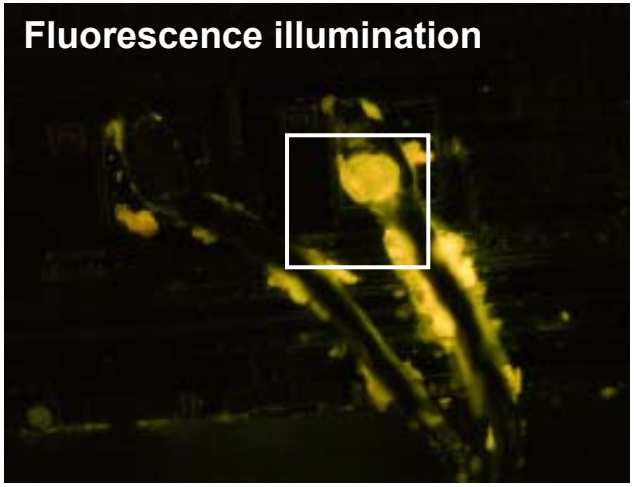
Zn zincite (ZnO)

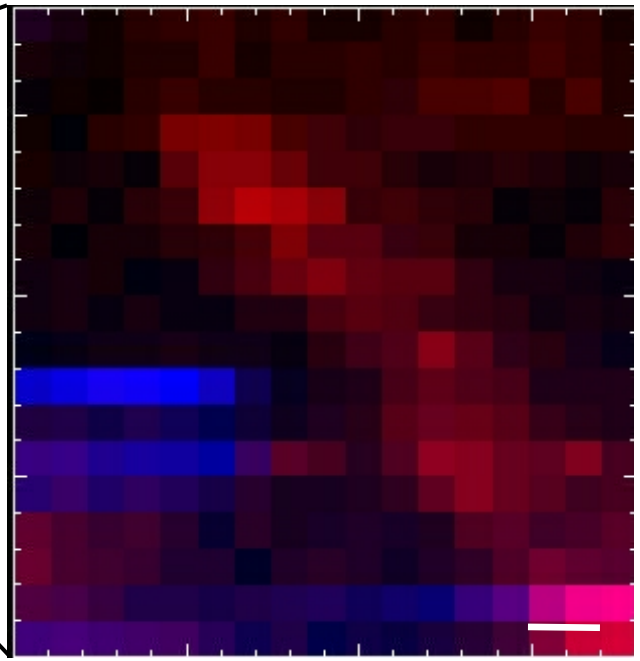
Fe franklinite ($ZnFeMnO_4$)

Scale bar is 20 microns

Contaminants on Computer Chips







chloride

gold

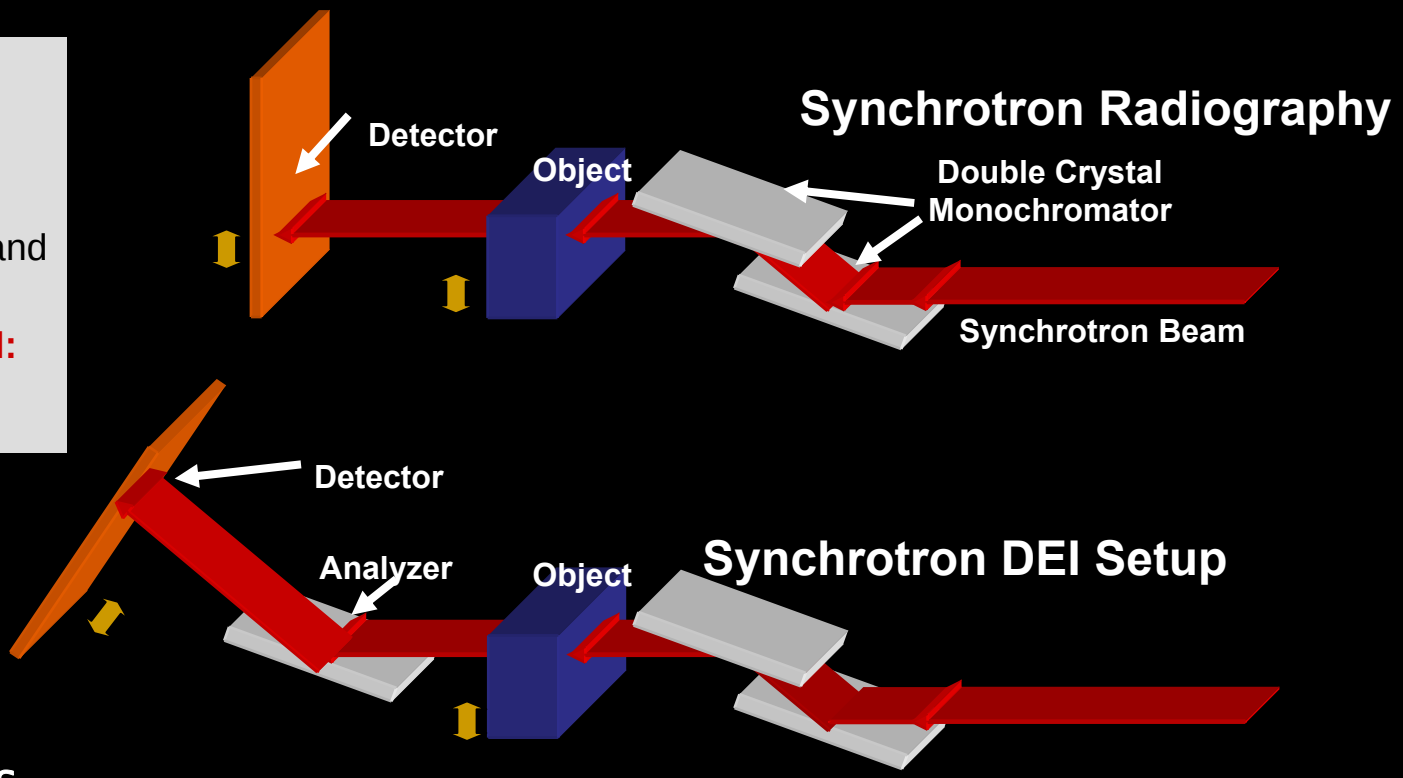
Scale bar is 20 microns

Diffraction-Enhanced Imaging

ENERGY RANGE:
- 10 – 30 keV

CONTRAST:
- absorption, refraction, and scatter rejection

SPATIAL RESOLUTION:
- 10 μm



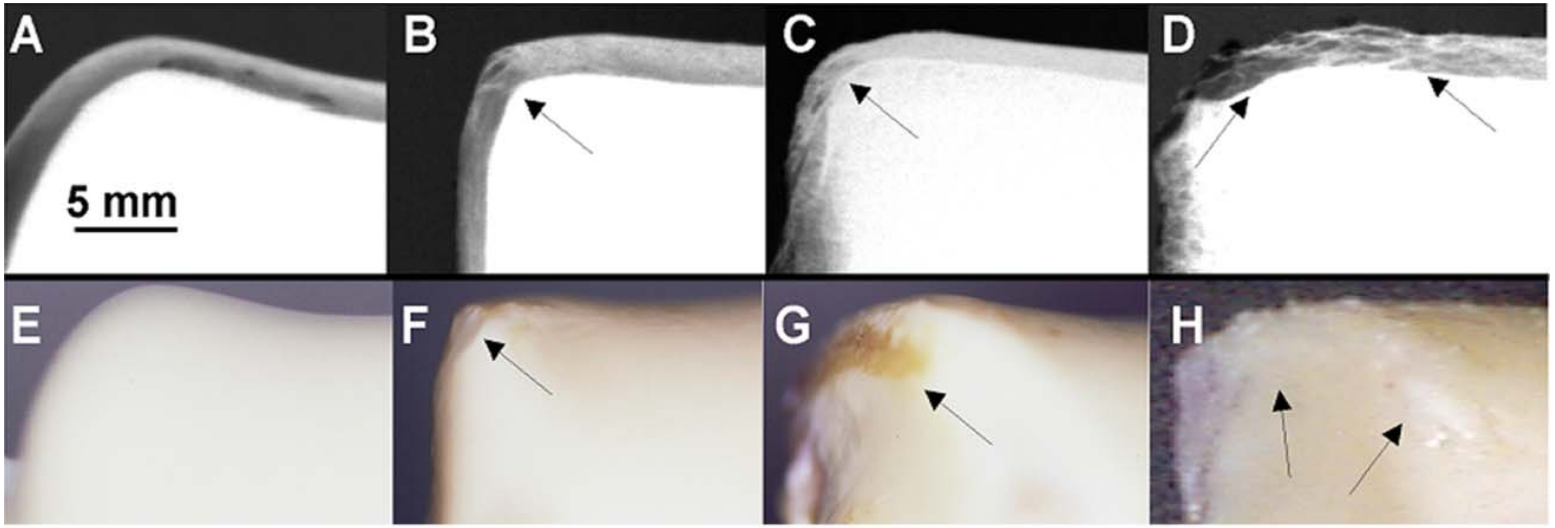
UNIQUE CAPABILITIES:

- Does not rely on the absorption of x-rays for the image contrast.
- Better contrast for soft-tissue imaging than conventional radiography
- Spatial resolution depends on source brightness and detector resolution, not absorption.
- At higher x-ray energies, the radiation dose to the subject is reduced significantly
- Examples: lung tissue substructure, calcifications and malignancies in breast tissue, torn cartilage in osteoarthritis, and ligaments and tendons in knee joints.

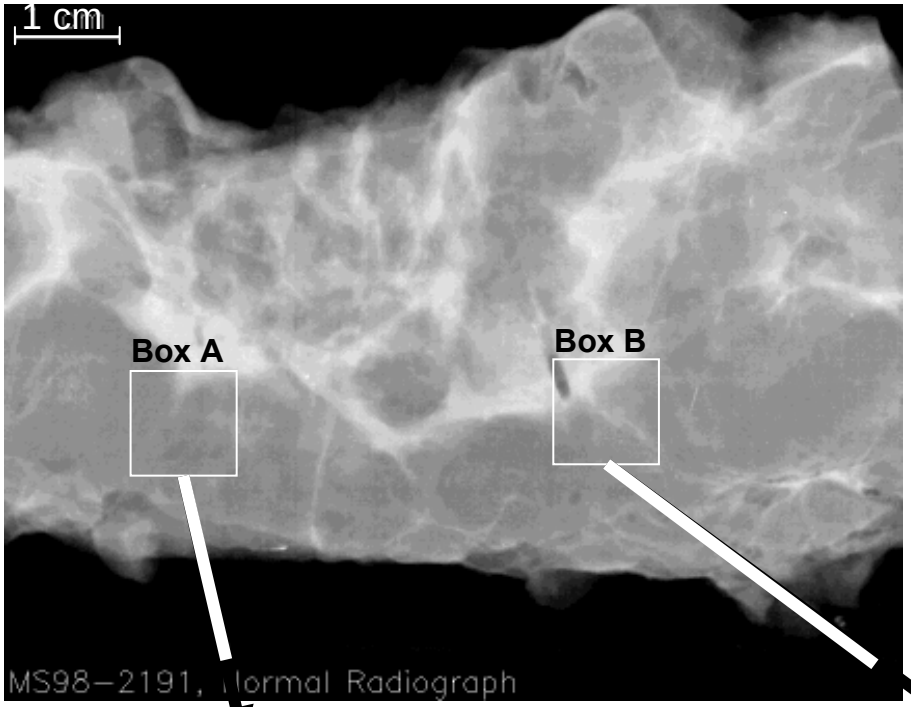
Soft-Tissue Imaging: Osteoarthritis

DEI

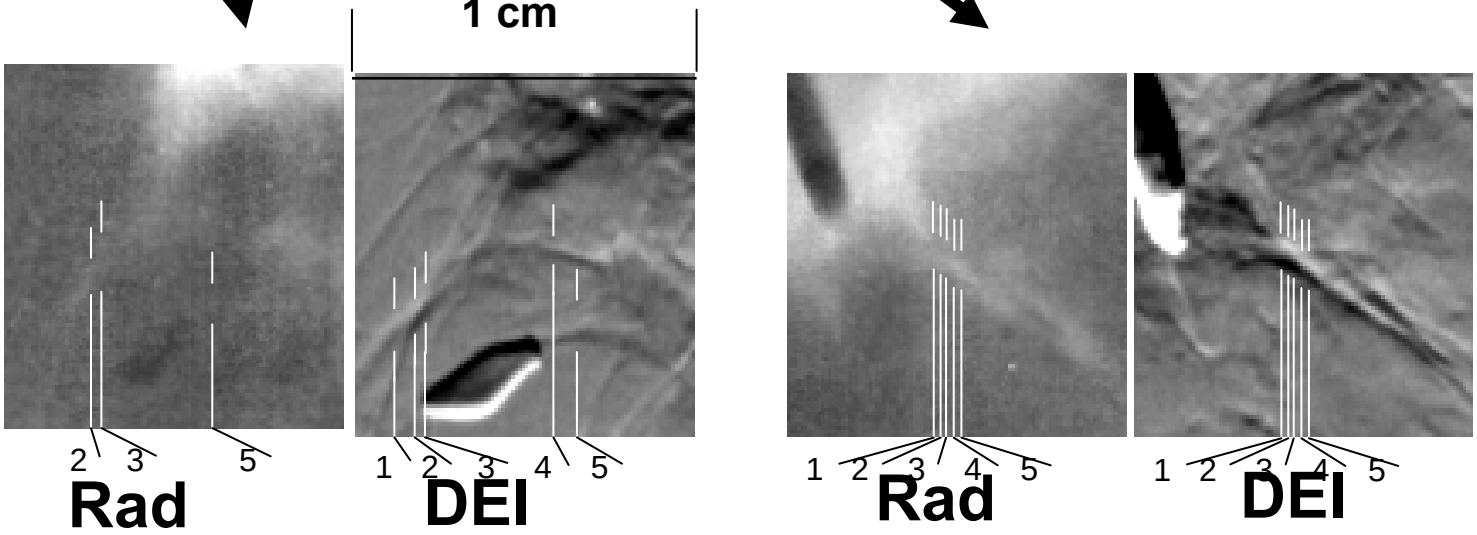
Optical



Spiculations in Breast Cancer



- Spiculations are due to cancer itself or the response of the host to the cancer
- Contrast quantified by measuring intensity change across spiculations
- DEI has 8 – 33 times greater contrast



Hasnah et. al., *Phys. Med. Bio.* 2006

Infrared Microspectroscopy & Imaging

ENERGY RANGE:

- 4000 – 500 cm^{-1} (2 – 20 μm)

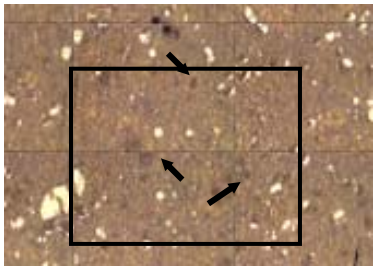
CONTRAST:

- organic composition by absorption of vibrational modes

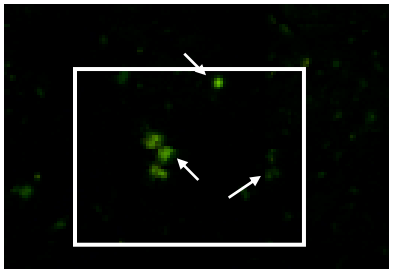
SPATIAL RESOLUTION:

- 2 – 20 μm (diffraction-limited)

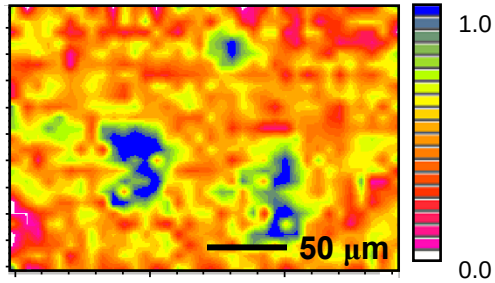
Visible light image



Fluorescence image



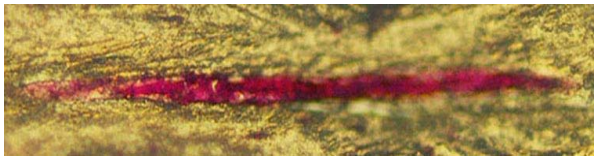
Infrared image

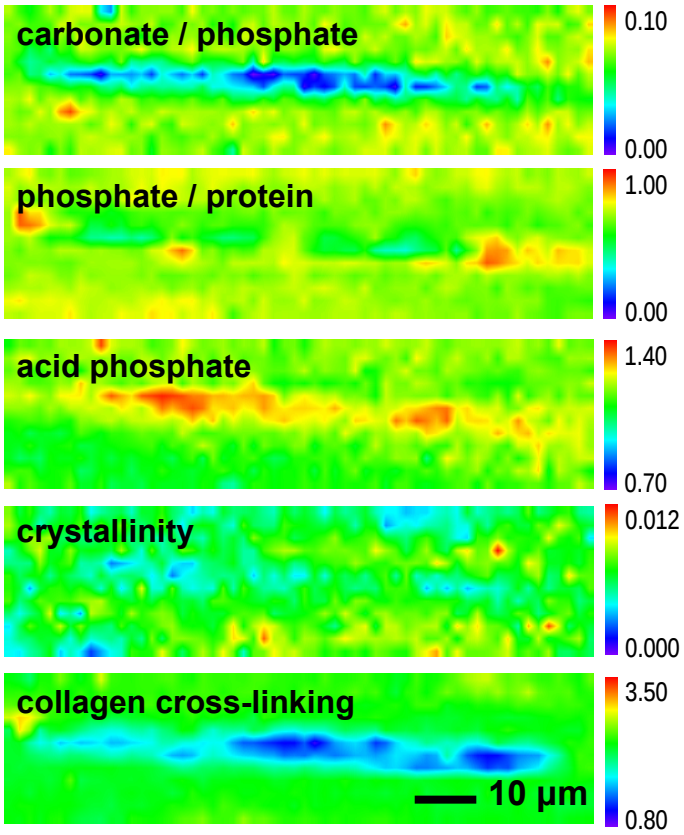


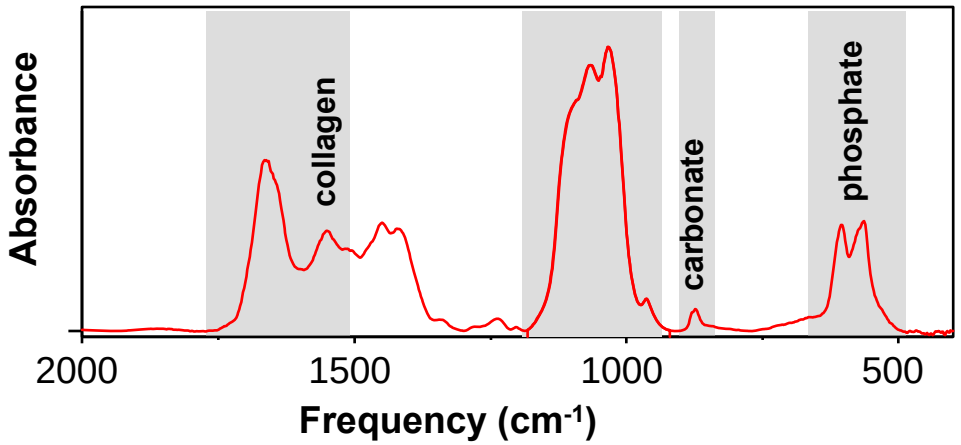
UNIQUE CAPABILITIES:

- High brightness of the synchrotron source permits diffraction-limited spatial resolution. Conventional IR imaging is throughput-limited to ~30 μm.
- Cellular-level imaging is with chemical sensitivity provides a direct indication of sample biochemistry.
- Instrument modified for simultaneous epi-fluorescence microscopy and IR imaging
- Examples: aggregates of misfolded proteins, e.g. amyloid plaques and infectious prion proteins; spectral evidence of cervical cancer, heart disease, and bone diseases such as osteoarthritis, osteoporosis, and osteogenesis imperfecta; contaminants in human tissue, such as silicone in breast tissue and narcotics in human hair

Microdamage Composition in Bone



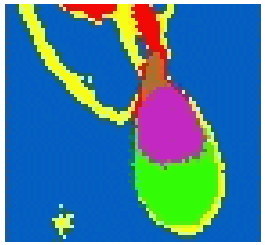


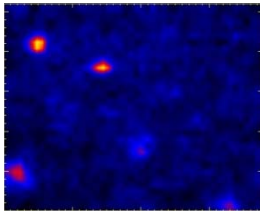


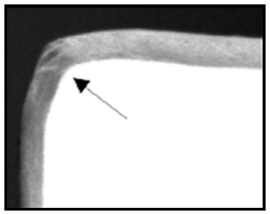
- Bisphosphonates are current treatment for osteoporosis.
- Microdamage increases in bisphosphonate-treated bone.
- Bone composition is different in microcrack region.
- With too much accumulation of microdamage, the quality of bone may be reduced.

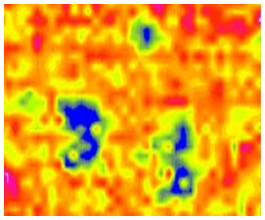
M. Ruppel, et al. *Bone*, 39(2): 318 (2006).

Who to Contact









Soft X-Ray Microscopy:

- Chris Jacobsen (Stony Brook University)
  - Beamline X1A1
  - life, environmental, polymer sciences

X-Ray Fluorescence Microprobe

- Tony Lanzirotti (University of Chicago)
  - Beamline X26A
  - Environmental, geosciences
- Paul Northrup (BNL-Enviro)
  - Beamline X27A
  - Environmental, geosciences
- Randy Smith (BNL-NSLS)
  - Beamline X27A
  - geosciences, life sciences

Diffraction Enhanced Imaging (DEI)

- Zhong Zhong (BNL-NSLS)
  - Beamline X15B
  - Life, environmental, materials science

Infrared Microspectroscopy & Imaging

- Lisa Miller (BNL-NSLS)
  - Beamline U10B
  - Life, environmental sciences
- Larry Carr (BNL-NSLS)
  - Beamline U10A
  - Materials science, condensed matter physics